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(Rev. 9-2001)

U.S. DEPARTMENT OF COMMERCE PATENT AND TRADEMARK OFFICE

ATTORNEY'S DOCKET NUMBER

**TRANSMITTAL LETTER TO THE UNITED STATES  
DESIGNATED/ELECTED OFFICE (DO/EO/US)  
CONCERNING A FILING UNDER 35 U.S.C. 371**

022650-690

U.S. APPLICATION NO. (If known, see 37 C.F.R. 1.5)

**10/070457**

INTERNATIONAL APPLICATION NO.  
PCT/FR00/02460

INTERNATIONAL FILING DATE  
September 7, 2000

PRIORITY DATE CLAIMED  
September 7, 1999

TITLE OF INVENTION

**METHOD OF CONTROLLING SOIL INSECTS WITH PHENYLPYRAZOLES**

APPLICANT(S) FOR DO/EO/US

**Jean-Michel GAULLIARD and Christian SEGAUD**

Applicant herewith submits to the United States Designated/Elected Office (DO/EO/US) the following items and other information:

1. ☒ This is a **FIRST** submission of items concerning a filing under 35 U.S.C. 371.
2. ☐ This is a **SECOND** or **SUBSEQUENT** submission of items concerning a filing under 35 U.S.C. 371.
3. ☒ This is an express request to begin national examination procedures (35 U.S.C. 371(f)). The submission must include items (5), (6), (9) and (21) indicated below.
4. ☒ The US has been elected by the expiration of 19 months from the priority date (Article 31).
5. ☒ A copy of the International Application as filed (35 U.S.C. 371(c)(2)), including the Request and International Search Report.
  - a. ☒ is attached hereto (required only if not communicated by the International Bureau).
  - b. ☐ has been communicated by the International Bureau.
  - c. ☐ is not required, as the application was filed in the United States Receiving Office (RO/US).
6. ☒ An English language translation of the International Application as filed (35 U.S.C. 371(c)(2)), including the Request and International Search Report.
  - a. ☒ is attached hereto.
  - b. ☐ has been previously submitted under 35 U.S.C. 154(d)(4).
7. ☒ Amendments to the claims of the International Application under PCT Article 19 (35 U.S.C. 371(c)(3))
  - a. ☐ are attached hereto (required only if not communicated by the International Bureau).
  - b. ☐ have been communicated by the International Bureau.
  - c. ☐ have not been made; however, the time limit for making such amendments has NOT expired.
  - d. ☒ have not been made and will not be made.
8. ☐ An English language translation of the amendments to the claims under PCT Article 19 (35 U.S.C. 371(c)(3)).
9. ☐ An oath or declaration of the inventor(s) (35 U.S.C. 371(c)(4)).
10. ☐ An English language translation of the annexes to the International Preliminary Examination Report under PCT Article 36 (35 U.S.C. 371(c)(5)).

Items 11 to 20 below concern document(s) or information included:

11. ☐ An Information Disclosure Statement under 37 CFR 1.97 and 1.98.
12. ☐ An assignment document for recording. A separate cover sheet in compliance with 37 CFR 3.28 and 3.31 is included.
13. ☒ A **FIRST** preliminary amendment.
14. ☐ A **SECOND** or **SUBSEQUENT** preliminary amendment.
15. ☐ A substitute specification.
16. ☐ A change of power of attorney and/or address letter.
17. ☐ A computer-readable form of the sequence listing in accordance with PCT Rule 13ter.2 and 35 U.S.C. 1.821 - 1.825.
18. ☐ A second copy of the published international application under 35 U.S.C. 154(d)(4).
19. ☐ A second copy of the English language translation of the international application under 35 U.S.C. 154(d)(4).
20. ☒ Other items or information:

Separate Claim for Convention Priority and of Form PCT/IB/304 (in French).  
Form PCT/IB/308.  
International Preliminary Examination Report (Form PCT/IPEA/409) (in French and English).

The executed Declaration of the inventors will follow.



**21839**

U.S. APPLICATION NO. (If known, see 37 C.F.R. 1.51)		INTERNATIONAL APPLICATION NO		ATTORNEY'S DOCKET NUMBER	
10/070457		PCT/FR00/02460		022650-690	
21. <input checked="" type="checkbox"/> The following fees are submitted:			CALCULATIONS		PTO USE ONLY
Basic National Fee (37 CFR 1.492(a)(1)-(5)):					
Neither international preliminary examination fee (37 CFR 1.482) nor international search fee (37 CFR 1.445(a)(2)) paid to USPTO and International Search Report not prepared by the EPO or JPO . . . . . \$1,040.00 (960)					
International preliminary examination fee (37 CFR 1.482) not paid to USPTO but International Search Report prepared by the EPO or JPO . . . . . \$890.00 (970)					
International preliminary examination fee (37 CFR 1.482) not paid to USPTO but international search fee (37 CFR 1.445(a)(2)) paid to USPTO . . . . . \$740.00 (958)					
International preliminary examination fee (37 CFR 1.482) paid to USPTO but all claims did not satisfy provisions of PCT Article 33(1)-(4) . . . . . \$710.00 (956)					
International preliminary examination fee (37 CFR 1.482) paid to USPTO and all claims satisfied provisions of PCT Article 33(1)-(4) . . . . . \$100.00 (962)					
ENTER APPROPRIATE BASIC FEE AMOUNT =			\$	890.00	
Surcharge of \$130.00 (154) for furnishing the oath or declaration later than 20 <input type="checkbox"/> 30 <input checked="" type="checkbox"/> months from the earliest claimed priority date (37 CFR 1.492(e)).			\$	130.00	
Claims	Number Filed	Number Extra	Rate		
Total Claims	33 -20 =	13	X\$18.00 (966)	\$	234.00
Independent Claims	1 -3 =	0	X\$84.00 (964)	\$	0.00
Multiple dependent claim(s) (if applicable)			+ \$280.00 (968)	\$	0.00
TOTAL OF ABOVE CALCULATIONS =			\$	1,254.00	
Reduction for 1/2 for filing by small entity, if applicable (see below).			+	\$	0.00
SUBTOTAL =			\$	1,254.00	
Processing fee of \$130.00 (156) for furnishing the English translation later than 20 <input type="checkbox"/> 30 <input type="checkbox"/> months from the earliest claimed priority date (37 CFR 1.492(f)).			+	\$	
TOTAL NATIONAL FEE =			\$	1,254.00	
Fee for recording the enclosed assignment (37 CFR 1.21(h)). The assignment must be accompanied by an appropriate cover sheet (37 CFR 3.28, 3.31). \$40.00 (581) per property			+	\$	0.00
TOTAL FEES ENCLOSED =			\$	1,254.00	
			Amount to be refunded:	\$	
			charged:	\$	
a. <input type="checkbox"/> Small entity status is hereby claimed.					
b. <input checked="" type="checkbox"/> A check in the amount of \$ 1,254.00 to cover the above fees is enclosed.					
c. <input type="checkbox"/> Please charge my Deposit Account No. 02-4800 in the amount of \$_____ to cover the above fees. A duplicate copy of this sheet is enclosed.					
d. <input checked="" type="checkbox"/> The Commissioner is hereby authorized to charge any additional fees which may be required, or credit any overpayment to Deposit Account No. 02-4800. A duplicate copy of this sheet is enclosed.					
NOTE: Where an appropriate time limit under 37 CFR 1.494 or 1.495 has not been met, a petition to revive (37 CFR 1.137(a) or (b)) must be filed and granted to restore the application to pending status.					
SEND ALL CORRESPONDENCE TO:					
Mary Katherine Baumeister BURNS, DOANE, SWECKER & MATHIS, L.L.P. P.O. Box 1404 Alexandria, Virginia 22313-1404 (703) 836-6620					
			George F. Lesmes		
			NAME		
			19,995		
			REGISTRATION NUMBER		
			March 7, 2002		
			DATE		

Patent Application  
Attorney's Docket No. 022650-690

**IN THE UNITED STATES PATENT AND TRADEMARK OFFICE  
UNITED STATES ELECTED OFFICE (EO/US)  
UNDER THE PATENT COOPERATION TREATY**

In re Patent Application of	)	
	)	
Jean-Michel GAULLIARD et al	)	Group Art Unit:
	)	
International Application No.:	)	Examiner:
PCT/FR00/02460	)	
	)	
International Filing Date: September 7, 2000	)	
	)	
U.S. Application No.	)	
	)	
Date of Commencement of U.S. National	)	
Phase: March 7, 2002	)	
	)	
For: METHOD OF CONTROLLING	)	
SOIL INSECTS WITH	)	
PHENYLPYRAZOLES	)	

**PRELIMINARY AMENDMENT**

**BOX PCT**  
Assistant Commissioner for Patents  
Washington, D.C. 20231

Sir:

Prior to examination on the merits, please amend the accompanying application filed  
under 35 U.S.C. § 371 as follows:

**IN THE SPECIFICATION:**

Page 1, prior to line 3, add --BACKGROUND OF THE INVENTION--.

Page 1, after line 8, add --Description of Related Art--.

Page 2, after line 23, add --SUMMARY OF THE INVENTION--.

Page 5, after line 10, add --DESCRIPTION OF PREFERRED EMBODIMENTS--.

**IN THE CLAIMS:**

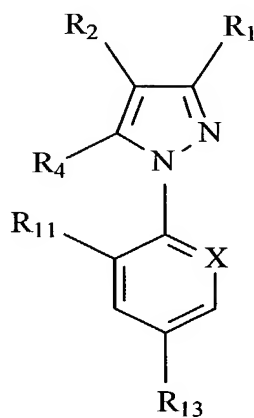
Page 17, at the top of the English translation of the published PCT application,  
amend the heading for the claims to read:

--WHAT IS CLAIMED IS:--

Please cancel Claims 1-17 and add the following new Claims 18-50:

--18. (New) Insecticidal compositions comprising:

(a) between 0.001 and 5 % of a compound of formula (I):



(I)

in which:

R<sub>1</sub> is a halogen atom or a CN group or a methyl group or a CH<sub>3</sub>CO group;

$R_2$  is  $S(O)_nR_3$ ;

$R_3$  is alkyl or haloalkyl;

$R_4$  represents a hydrogen or halogen atom, or an  $NR_5R_6$ ,  $S(O)_mR_7$ ,  $C(O)R_7$  or  $C(O)O-R_7$ , alkyl, haloalkyl or  $OR_8$  radical or an  $-N=C(R_9)(R_{10})$  radical;

$R_5$  and  $R_6$  independently represent a hydrogen atom or an alkyl, haloalkyl,  $C(O)$ alkyl or  $S(O)_rCF_3$  radical, or  $R_5$  and  $R_6$  can together form a divalent alkylene radical optionally interrupted by one or two divalent heteroatoms;

$R_7$  represents an alkyl or haloalkyl radical;

$R_8$  represents an alkyl or haloalkyl radical or a hydrogen atom;

$R_9$  represents an alkyl radical or a hydrogen atom;

$R_{10}$  represents a phenyl or heteroaryl group optionally substituted with at least one halogen atom or groups selected from OH, -O-alkyl, -S-alkyl, cyano or alkyl;

X represents a trivalent nitrogen atom or a  $C-R_{12}$  radical, the other three valences of the carbon atom forming part of the aromatic ring;

$R_{11}$  and  $R_{12}$  represent, independently of each other, a hydrogen or halogen atom;

$R_{13}$  represents a halogen atom or a haloalkyl, haloalkoxy,  $S(O)_qCF_3$  or  $SF_5$  group;

m, n, q, r represent, independently of each other, an integer equal to 0, 1 or 2;

with the proviso that when  $R_1$  is methyl, then  $R_3$  is haloalkyl,  $R_4$  is  $NH_2$ ,  $R_{11}$  is Cl,

$R_{13}$  is  $CF_3$  and X is N;

(b) between 0.05 and 10 % of at least one moisture-retaining agent; and

(c) between 40 and 99% of at least one vegetable meal.

19. (New) Insecticidal compositions according to Claim 18, wherein the vegetable meal is derived from the grinding of a cereal grain.
20. (New) Insecticidal compositions according to Claim 18, wherein the vegetable meal is a maize.
21. (New) Insecticidal compositions according to Claim 18, wherein the moisture-retaining agent is of an organic nature.
22. (New) Insecticidal compositions according to Claim 18, wherein the composition also comprises from 3 to 30 % of a sugar.
23. (New) Insecticidal compositions according to Claim 22, wherein the sugar is selected from mono, oligo- or polyorganosaccharides.
24. (New) Insecticidal compositions according to Claim 18, further comprising a preservative to prevent degradation of the meal.
25. (New) Insecticidal compositions according to Claim 18, further comprising one or more additives selected from colorings, attractants for pests, repellants for birds or animals which are useful or which should be protected, binding agents, agglomerating

agents, appetite-enhancing agents, agglutinating agents, gelling agents, swelling agents or antiadherent agents.

26. (New) Insecticidal compositions according to Claim 18, wherein the compound of formula (I) is 5-amino-3-cyano-1-[2,6-di-chloro-4-(trifluoromethyl)phenyl]-4-[(trifluoromethyl)-sulphinyl]-1H-pyrazole.

27. (New) Insecticidal compositions according to Claim 18, which are in the form of granules of a size between 0.1 mm and 3 cm.

28. (New) Insecticidal compositions according to Claim 18, wherein the compound of formula (I) is present in an amount of between 0.05 and 1 %.

29. (New) Insecticidal compositions according to Claim 28, wherein the compound of formula (I) is present in an amount of between 0.05 and 0.5 %.

30. (New) Insecticidal compositions according to Claim 18, wherein the moisture-retaining agent is present in an amount of between 0.1 and 5 %.

31. (New) Insecticidal compositions according to Claim 18, wherein the vegetable meal is present in an amount of between 50 and 98 %.

38. (New) Insecticidal compositions according to Claim 24, wherein the preservative is selected from sodium benzoate, 1,2-benziso-thiazolin-3-one, benzoic acid,



para-hydroxybenzoic acid and its esters and alkali or alkaline-earth metal salts,  
2-phenylphenol and its alkali or alkaline-earth metal salts, or para-nitrophenol.

39. (New) Insecticidal compositions according to Claim 27, wherein the granules are of a size between 0.5 and 4 mm and are water-insoluble.

40. (New) Insecticidal compositions according to Claim 18, further including from 0.5 to 30 % of at least one disintegrating agent.

41. (New) Insecticidal compositions according to Claim 40, wherein the disintegrating agent is present in an amount of 1 to 20 % and is selected from starch, sodium carboxymethyl starch, microcrystalline cellulose; modified celluloses; bentonite, aluminium or magnesium silicate; sodium polynaphthalenesulphonate, sodium dodecylbenzenesulphonate, sodium dioctylsulphosuccinate, lignin sulphonate; a saccharide derivative; or a cross-linked derivative of polyvinylpyrrolidone.

42. (New) A method of controlling insects which comprises applying an effective quantity of a composition according to Claim 18 in the form of granules having a size of between 0.2 mm and 2 cm over or into the soil in an area which has to be cultivated.

43. (New) A method according to Claim 42, wherein the effective quantity is selected to provide a dosage which is nonlethal through contact but lethal through ingestion.

50. (New) A method according to Claim 49, wherein the effective quantity is between 3 and 40 g/ha.

Application No.  
Attorney's Docket No. 022650-690  
Page 9

**IN THE ABSTRACT OF THE DISCLOSURE:**

Please add the abstract presented on the accompanying separate page 22 to replace the abstract appearing on the cover of the English translation of the published version of the PCT application.

## REMARKS

Entry of the foregoing and prompt and favorable consideration of the subject application, in light of the following remarks, are respectfully requested.

The foregoing amendments have been made to place the claims in better form for examination and eliminate multiple dependent claim terminology. Claims 1-17 have been canceled without prejudice or disclaimer to the subject matter recited therein and new Claims 18-50 have been added. Support for Claims 18-50 can be found in canceled Claims 1-17 and throughout the specification of the subject application. No new matter has been added by the foregoing amendment.

In the event that there are any questions relating to this Preliminary Amendment, or the application in general, it would be appreciated if the Examiner would telephone the undersigned concerning such questions so that prosecution of this application may be expedited.

Respectfully submitted,

BURNS, DOANE, SWECKER & MATHIS, L.L.P.

By:

George F. Lesmes  
Registration No. 19,995

**Post Office Box 1404  
Alexandria, Virginia 22313-1404  
(703) 836-6620**

**Date: March 7, 2002**

10/070457

JC13 Rec'd PCT/PTO 07 MAR 2002

Application No.  
Attorney's Docket No. 022650-690  
Page 1

**Attachment to Preliminary Amendment dated March 7, 2002**

**Marked-up Claims**

Claims heading on page 17:

[CLAIMS]WHAT IS CLAIMED IS:

# ABSTRACT OF THE DISCLOSURE

The invention concerns insecticide compositions comprising:

- (a) between 0.001 and 5 %, preferably between 0.05 and 1 % of a  
5 compound of the type 1-phenyl pyrazole, in particular 1-[2,6-Cl<sub>2</sub>4-CF<sub>3</sub> phenyl]3-CN 4-[SO-CF<sub>3</sub>]5-NH<sub>2</sub> pyrazole; and
- (b) between 0.05 and 10 %, preferably between 0.1 and 5 % of one (or  
several) moisture retaining agent of the organic type; and
- (c) between 40 and 99 %, preferably between 50 and 98 % (and more  
10 preferably between 70 and 97 %) of vegetable flour.

The invention also concerns a method for fighting insects using said compositions, in particular against click beetles.

WO 01/17354

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PCT/FR00/02460

**Method of controlling soil insects with phenylpyrazoles**

The subject of the present invention is novel compositions intended for controlling soil insects in  
5 their various developmental forms, and in particular compositions useful for controlling click beetles. The invention also relates to a method of control using the said compositions.

Insecticidal compounds of the phenylpyrazole  
10 type which can be used in controlling insects are known in particular from patent applications EP 295117, WO 87/3781, 93/6089 and 94/21606. Patent applications EP 295117 and 836386 also mention compositions comprising from 0.01% to 5% of such active substances.

15 Click beetles constitute a family of insects which are particularly harmful for certain crops, more particularly for maize, beet, sunflower, potato and rape crops. Their harmful character is all the more marked since the larval forms of click beetles can  
20 remain for very long periods in the soil, extending up to 5 years.

Baits have indeed been proposed for various sorts of insects, as well as formulas which can be consumed by ingestion, but these formulas are not  
25 necessarily active for all the types of insect and the need remains to find insecticidal forms or formulations which are particularly effective for the most diverse



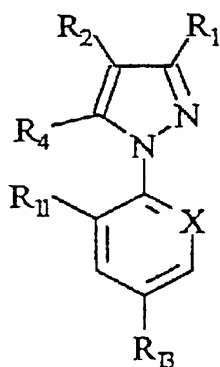


specified that the percentages indicated in the present text are weight/weight percentages, unless otherwise indicated.

The subject of the present invention is therefore, firstly, insecticidal compositions comprising:

- a) between 0.001 and 5%, preferably between 0.05 and 1% and still more advantageously between 0.05 and 0.5% of the compound of formula (I):

10



(I)

in which:

R<sub>1</sub> is a halogen atom or a CN group or a methyl group or a CH<sub>3</sub>CO group;

15 R<sub>2</sub> is S(O)<sub>n</sub>R<sub>3</sub>;

R<sub>3</sub> is alkyl or haloalkyl;

R<sub>4</sub> represents a hydrogen or halogen atom, or an NR<sub>5</sub>R<sub>6</sub>, S(O)<sub>m</sub>R<sub>7</sub>, C(O)R<sub>7</sub> or C(O)O-R<sub>7</sub>, alkyl, haloalkyl or OR<sub>8</sub> radical or an -N=C(R<sub>9</sub>)(R<sub>10</sub>) radical;

20 R<sub>5</sub> and R<sub>6</sub> independently represent a hydrogen atom or an alkyl, haloalkyl, C(O)alkyl or S(O)<sub>r</sub>CF<sub>3</sub>

R<sub>7</sub> represents an alkyl or haloalkyl radical;

R<sub>9</sub> represents an alkyl radical or a hydrogen

R<sub>10</sub> represents a phenyl or heteroaryl group

X represents a trivalent nitrogen atom or a radical, the other three valencies of the carbon

$R_{11}$  and  $R_{12}$  represent, independently of each

R<sub>13</sub> represents a halogen atom or a haloalkyl,

m, n, q, r represent, independently of each

with the proviso that when R<sub>1</sub> is methyl, then  
alkyl, R<sub>4</sub> is NH<sub>2</sub>, R<sub>11</sub> is Cl, R<sub>13</sub> is CF<sub>3</sub> and X is

- b) between 0.05 and 10%, preferably between 0.1% and 5% by weight based on the total weight of one (or more) moisture-retaining agents, or  
- c) by a moisture-retaining agent of an organic nature.

nature; and

- c) between 40 and 99%, preferably between 50 and 98% (and more preferably between 70 and 97%) of vegetable meal.

5           The alkyl radicals of the definition of formula (I) generally comprise from 1 to 6 carbon atoms. The ring formed by the divalent alkylene radical representing  $R_5$  and  $R_6$  as well as by the nitrogen atom to which  $R_5$  and  $R_6$  are attached is generally a 5-, 6- or  
10 7-membered ring.

          The compound of formula (I) may be prepared according to one of the methods described in patent applications WO 87/3781, 93/6089, 94/21606, EP 295117 or alternatively by another method within the general  
15 knowledge of persons skilled in the art competent in chemical synthesis. This compound is generally designated in the present text by the term active substance.

          Among the vegetable meals which can be used,  
20 there may be mentioned the meals derived from the grinding of cereal grains such as wheat, barley, rye, triticale, oats, or also rice, sorghum, soyabean, maize, the preferred meal being that based on maize. A mixture of these vegetable meals can also be envisaged  
25 in the context of the present invention.

          Among the moisture-retaining agents of an organic nature, there may be mentioned the

macromolecular hydrophilic derivatives of plant origin, and in particular the cellulosic hydrophilic derivatives, and more particularly cellulose, but also one or more disintegrating agents. It may be

5 advantageous to use these compounds in particular when meals such as hard wheat meals are used in the granules. Disintegrating agents include: starch, sodium carboxymethyl starch, cellulose such as microcrystalline cellulose; modified celluloses such as

10 sodium carboxymethylcellulose; bentonite, aluminium and magnesium silicate; sodium polynaphthalenesulphonate, sodium dodecylbenzenesulphonate, sodium dioctylsulphosuccinate, lignin sulphonate; a saccharide derivative such as lactose, fructose, sucrose,

15 mannitol, dextrose; a cross-linked derivative of polyvinylpyrrolidone. When a disintegrating agent is used, the composition according to the invention, may contain from 0.5 to 30%, and preferably from 1 to 20%, by weight of the dry substance, of the said agent(s).

20 According to a variant of the composition according to the invention, the composition also comprises from 3 to 30%, preferably from 4 to 20% of sugars. The sugars are chosen in particular from mono-, oligo- or polyorganosaccharides, especially from

25 sucrose, lactose, fructose, dextrose, glucose or alternatively molasses or honey.

The compositions which are the subject of the

According to a particularly advantageous variant of the invention, the compound of formula (I) used in the invention is 5-amino-3-cyano-1-[2,6-di-  
25 chloro-4-(trifluoromethyl)phenyl]-4-[(trifluoromethyl)-sulphanyl]-1*H*-pyrazole, to which reference is made in the examples under the term "fipronil".



to a method of protecting cereal, preferably maize or beet or sunflower or potato or rape, crops. The application of the formulations according to the invention takes place advantageously before sowing the  
5 said crop, or simultaneously with this sowing.

The invention also relates to a method of controlling insects, especially click beetles, characterized in that an effective quantity of one of the compositions according to the invention is applied  
10 over or into the soil (preferably into the soil) where they are present or are likely to be present.

As effective quantity, quantities of composition corresponding to a dose of compound of formula (I) of between 1 and 50 g/ha, preferably  
15 between 3 and 40 g/ha are often used.

A specific characteristic of the method of controlling insects according to the invention consists in the application, over or into the soil, of a composition providing a dose which is nonlethal through  
20 contact but lethal through ingestion.

In other words, in the specific case of click beetles, the method consists in killing the click beetles by application of a dose which is nonlethal through contact but lethal through ingestion. A  
25 hypothesis for the good efficacy of the method of treatment according to the invention, which makes it possible to greatly reduce the applicable doses of

compounds of formula I in particular, is based on the fact that once the bait according to the invention has caused the death of a click beetle, the latter can itself serve as bait for other click beetles, which therefore also ingest a product (dead click beetle) containing the insecticide.

For the purposes of the present text, the words insecticide and insect should be taken in their broad ordinary sense and not in their strictly scientific (zoological) sense. Accordingly, the term insect is understood to mean any animal of a very small size such as arthropods (insects in the strict and zoological sense, arachnids, myriapods) and nematodes.

As soil insects against which the invention  
15 is particularly effective, there may be mentioned for  
example:

The **Coleoptera** (wireworms (*Agriotes* spp.), false wireworms, white grubs) such as for example:

Agriotes lineatus (European click beetle,  
20 Elateridae),

*Agriotes sordidus* (European click beetle,  
Elateridae),

*Agriotes obscurus* (European click beetle,  
Elateridae),

25        *Agriotes sputator* (European click beetle,  
Elateridae),

*Athous* spp. (Elateridae),





The following examples illustrate the invention without however constituting a limitation thereto. In these examples, the compound of formula (I) used is fipronil.

5                      Example 1:

A surface of 0.1 ha is sown with maize at the rate of about 8000 untreated seeds. This surface is divided into 40-m<sup>2</sup> plots.

At the same time as the sowing, there are  
10 incorporated into the soil, in the sowing row, 2 mm  
granules containing a composition consisting of:

0.25% of fipronil,  
93.5% of maize meal,  
2% of cellulose,  
4% of lactose,  
0.2% of para-nitrophenol,  
0.05% of pigment blue 15.3.

The quantities of granules thus spread vary from 2.5 to 10 kg per hectare. Untreated plots are kept to serve as control and to verify the extent of the damage by the insects. Likewise, the plots will be treated with a commercial insecticide which is reputed effective and called reference. Each modality is repeated four times.

25                    Approximately 20 days after sowing, the maize  
plants which have emerged are counted.

In the locality of St Hilaire (30), 22 days

after sowing, 19 plants are observed in the furrow of the 4 untreated plots per 10 metres of furrow. In the plots treated according to the invention with the dose of fipronil of 6 g per hectare, 49 plants are observed 5 per 10 metres of furrow.

With the dose of fipronil of 12.5 g per hectare, 48 to 50.5 plants are observed per 10 metres of furrow.

With the dose of fipronil of 25 g per 10 hectare, 45.5 to 51.3 plants are observed per 10 metres of furrow.

With the dose of fipronil of 50 g per hectare, 49 plants are observed per 10 metres of furrow.

15 To obtain the same result with a conventional granule (clay carrier), 200 g of fipronil have to be provided per hectare.

The soil is dug out and scraped in order to capture and identify the insects responsible for the 20 damage; a large presence of larvae of click beetles of the genus Agriotes, in particular Agriotes sordidus, is observed for the untreated control.

#### Example 2:

A surface of 1 ha is sown with maize at the 25 rate of about 98,100 untreated seeds. This surface is divided into 27-m<sup>2</sup> plots.

At the same time as the sowing, there are

With the dose of fipronil of 25 g per

With the dose of fipronil of 50 g per hectare, 49.8 plants are observed per 10 metres of furrow.

The soil is dug out and scraped in order to capture and identify the insects responsible for the damage; a large presence of larvae of click beetles of the genus *Agriotes*, in particular *Agriotes lineatus*.

15           At the same time as the sowing, there are  
incorporated into the soil, in the sowing row, granules  
according to the invention consisting of:

20                    945.5 g/kg of a) **maize meal (granules A)** or  
b) **rice meal (granules B)**,

2 g/kg of para-nitrophenol.

The efficacy against Agriotes (click  
25 beetles), 90 days after sowing, of the two granules A  
and B above, used at the doses of 5 and 2.5 g of  
fipronil per hectare, and of the same fipronil used by

spraying at the doses of 50 and 25 g/ha (from Regent® 800WG) (compound C), was compared.

The following results, expressed as number of holes per 10 tubers (= N) are then observed:

5

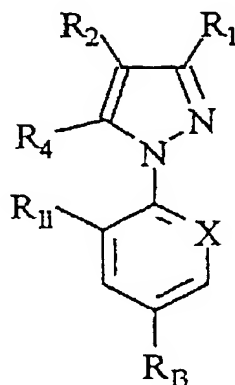
Product	A	A	B	B	C	C	Control*
Dose (g.ha)	2.5	5	2.5	5	25	50	-
N	6	1	7	3	6	1	14

\* untreated control.

This result indeed shows the good control of click beetles which is obtained by the granules according to the invention which give the same result as Regent® 800WG **but with a dose reduced by a factor of 10.**

CLAIMS

1. Insecticidal compositions comprising:  
 - a) between 0.001 and 5%, preferably  
 between 0.05 and 1% and still more advantageously  
 5 between 0.05 and 0.5% of a compound of formula (I):



(I)

in which:

10 R<sub>1</sub> is a halogen atom or a CN group or a methyl group or a CH<sub>3</sub>CO group;

R<sub>2</sub> is S(O)<sub>n</sub>R<sub>3</sub>;

R<sub>3</sub> is alkyl or haloalkyl;

R<sub>4</sub> represents a hydrogen or halogen atom, or  
 15 an NR<sub>5</sub>R<sub>6</sub>, S(O)<sub>m</sub>R<sub>7</sub>, C(O)R<sub>7</sub> or C(O)O-R<sub>7</sub>, alkyl, haloalkyl or OR<sub>8</sub> radical or an -N=C(R<sub>9</sub>)(R<sub>10</sub>) radical;

R<sub>5</sub> and R<sub>6</sub> independently represent a hydrogen atom or an alkyl, haloalkyl, C(O)alkyl or S(O)<sub>r</sub>CF<sub>3</sub> radical, or R<sub>5</sub> and R<sub>6</sub> can together form a divalent  
 20 alkylene radical which may be interrupted by one or two

divalent heteroatoms such as oxygen or sulphur;

$R_7$  represents an alkyl or haloalkyl radical;

$R_8$  represents an alkyl or haloalkyl radical or a hydrogen atom;

5  $R_9$  represents an alkyl radical or a hydrogen atom;

$R_{10}$  represents a phenyl or heteroaryl group optionally substituted with one or more halogen atoms or groups such as OH, -O-alkyl, -S-alkyl, cyano or  
10 alkyl;

$R_{11}$  and  $R_{12}$  represent, independently of each other, a hydrogen or halogen atom;

$R_{13}$  represents a halogen atom or a haloalkyl, haloalkoxy,  $S(O)_qCF_3$  or  $SF_5$  group;

15  $m$ ,  $n$ ,  $q$ ,  $r$  represent, independently of each other, an integer equal to 0, 1 or 2;

with the proviso that when  $R_1$  is methyl, then  $R_3$  is haloalkyl,  $R_4$  is  $NH_2$ ,  $R_{11}$  is Cl,  $R_{13}$  is  $CF_3$  and  $X$  is N;

20 - b) between 0.05 and 10%, preferably between 0.1 and 5% of one (or more) moisture-retaining agents, preferably a moisture-retaining agent of an organic nature; and

- c) between 40 and 99%, preferably  
25 between 50 and 98% (and more preferably between 70 and 97%) of vegetable meal.

2. Insecticidal compositions according to





5 2-phenylphenol and its alkali or alkaline-earth metal salts, in particular the sodium salt, para-nitrophenol.

15            9.    Insecticidal compositions according to  
one of Claims 1 to 8, characterized in that the  
compound of formula (I) is 5-amino-3-cyano-1-[2,6-di-  
chloro-4-(trifluoromethyl)phenyl]-4-[(trifluoromethyl)-  
sulphinyl]-1*H*-pyrazole.

11. Method of controlling insects,

characterized in that an effective quantity of a composition according to one of Claims 1 to 10 in the form of granules having a size of between 0.2 mm and 2 cm is applied over or into the soil (preferably into the soil) of the area which has to be cultivated.

12. Method of protecting crops according to Claim 11, characterized in that a compound of formula I is used.

13. Method according to either of Claims 11 and 12, characterized in that cereal, preferably maize or beet or sunflower or potato or rape, crops are protected.

14. Method according to one of Claims 11 to 13, characterized in that an effective quantity of one of the compositions according to the invention is used for controlling insects, especially click beetles.

15. Method according to one of Claims 12 to 14, characterized in that the effective quantity of composition corresponds to a dose of compound of formula (I) of between 1 and 50 g/ha, preferably between 3 and 40 g/ha.

16. Method of controlling insects which consists in the application, over or into the soil, of a composition providing a dose which is nonlethal through contact but lethal through ingestion.

17. Method according to Claim 16, applicable to click beetles.

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LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NO,  
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LS, MW, MZ, SD, SL, SZ, TZ, UG, ZW), brevet eurasien  
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Publiée:

— Avec rapport de recherche internationale.

En ce qui concerne les codes à deux lettres et autres abrégia-  
tions, se référer aux "Notes explicatives relatives aux codes et  
abrégiactions" figurant au début de chaque numéro ordinaire de  
la Gazette du PCT.

(54) Title: METHOD FOR FIGHTING SOIL INSECTS WITH PHENYL-PYRAZOLES

(54) Titre: PROCEDE DE LUTTE CONTRE LES INSECTES DU SOL AVEC DES PHENYL-PYRAZOLES

(57) Abstract: The invention concerns insecticide compositions comprising: a) between 0.001 and 5 %, preferably between 0.05 and 1 % of a compound of the type 1-phenyl pyrazole, in particular 1-[2,6-Cl<sub>2</sub>4-CF<sub>3</sub> phenyl] 3-CN 4-[SO-CF<sub>3</sub>] 5-NH<sub>2</sub> pyrazole; and b) between 0.05 and 10 %, preferably between 0.1 and 5 % of one (or several) moisture retaining agent of the organic type; and c) between 40 and 99 %, preferably between 50 and 98 % (and more preferably between 70 and 97%) of vegetable flour. The invention also concerns a method for fighting insects using said composition, in particular against click beetles.

(57) Abrégé: Compositions insecticides comprenant: a) entre 0,001 et 5 %, de préférence entre 0,05 et 1 % d'un composé de type 1-phényl pyrazole, notamment le 1-[2,6-Cl<sub>2</sub>4-CF<sub>3</sub>phényl]3-CN4-[SO-CF<sub>3</sub>]5-NH<sub>2</sub> pyrazole, et; b) entre 0,05 et 10 %, de préférence entre 0,1 et 5 % d'un (ou plusieurs) agent rétenteur d'humidité, de préférence un agent rétenteur d'humidité de nature organique, et; c) entre 40 et 99 %, de préférence entre 50 et 98 % (et plus préférentiellement entre 70 et 97 %) de farine végétale. Procédé de lutte contre les insectes mettant en oeuvre cette composition, notamment contre les taupins.

WO 01/17354 A1

**COMBINED DECLARATION AND POWER OF ATTORNEY  
FOR UTILITY OR DESIGN PATENT APPLICATION**

As a below named inventor, I hereby declare that:

My residence, post office address and citizenship are as stated below next to my name;

I believe I am the original, first and sole inventor (if only one name is listed below) or an original, first and joint inventor (if plural names are listed below) of the subject matter which is claimed and for which a patent is sought on the invention entitled:

**METHOD OF CONTROLLING SOIL INSECTS WITH PHENYLPYRAZOLES**

the specification of which (check only one item below):

- ☐ is attached hereto.
- ☐ was filed as United States application  
Number \_\_\_\_\_ on \_\_\_\_\_  
and was amended \_\_\_\_\_ on \_\_\_\_\_ (if applicable).
- ☒ was filed as PCT international application  
Number PCT/FR00/02460 on September 7, 2000  
and was amended \_\_\_\_\_ on \_\_\_\_\_ (if applicable).

I hereby state that I have reviewed and understand the contents of the above-identified specification, including the claims, as amended by any amendment referred to above.

I acknowledge the duty to disclose to the Office all information known to me to be material to patentability as defined in Title 37, Code of Federal Regulations, §1.56.

I hereby claim foreign priority benefits under Title 35, United States Code, §§119 (a)-(d), 172 or 365 of any foreign application(s) for patent or inventor's certificate or of any PCT international application(s) designating at least one country other than the United States of America listed below and have also identified below any foreign application(s) for patent or inventor's certificate or any PCT international application(s) designating at least one country other than the United States of America filed by me on the same subject matter having a filing date before that of the application(s) of which priority is claimed:

PRIOR FOREIGN/PCT APPLICATION(S) AND ANY PRIORITY CLAIMS UNDER 35 U.S.C. §§119(a)-(d), 172 or 365:					
COUNTRY (if PCT, indicate "PCT")	APPLICATION NUMBER	DATE OF FILING (day, month, year)	PRIORITY CLAIMED UNDER 35 U.S.C. §§119, 172 or 365		
FRANCE	99/11312	07 September 1999	X	Yes	No
				Yes	No
				Yes	No
				Yes	No
				Yes	No

**COMBINED DECLARATION AND POWER OF ATTORNEY  
FOR UTILITY OR DESIGN PATENT APPLICATION**

As a below named inventor, I hereby declare that:

My residence, post office address and citizenship are as stated below next to my name;

I believe I am the original, first and sole inventor (if only one name is listed below) or an original, first and joint inventor (if plural names are listed below) of the subject matter which is claimed and for which a patent is sought on the invention entitled:

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- ☐ is attached hereto.
- ☐ was filed as United States application  
Number \_\_\_\_\_ on \_\_\_\_\_  
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FRANCE	99/11312	07 September 1999	X Yes	No
			Yes	No
			Yes	No
			Yes	No
			Yes	No

Combined Declaration and Power of Attorney  
for Utility or Design Patent Application  
Attorney's Docket No. 022650-690

Page 2 of 2

I hereby appoint the following attorneys and agent(s) to prosecute said application and to transact all business in the Patent and Trademark Office connected therewith and to file, prosecute and to transact all business in connection with international applications directed to said invention:

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Platon N. Mandros	22,124	Teresa Stanek Rea	30,427	Ronni S. Jillions	31,979
Benton S. Duffett, Jr.	22,030	Robert E. Krebs	25,885	Harold R. Brown III	36,341
Norman H. Stepno	22,716	William C. Rowland	30,888	Allen R. Baum	36,086
Ronald L. Grudziecki	24,970	T. Gene Dillahunt	25,423	Brian P. O'Shaughnessy	32,747
Frederick G. Michaud, Jr.	26,003	Patrick C. Keane	32,858	Kenneth B. Leffler	36,075
Alan E. Kopecki	25,813	B. Jefferson Boggs, Jr.	32,344	Fred W. Hathaway	32,236
Regis E. Slutter	26,999	William H. Benz	25,952	Wendi L. Weinstein	34,456
Samuel C. Miller, III	27,360	Peter K. Skiff	31,917	Mary Ann Dillahunt	34,576
Robert G. Mukai	28,531	Richard J. McGrath	29,195	Donna M. Meuth	36,607
George A. Hovanec, Jr.	28,223	Matthew L. Schneider	32,814	Mark R. Kresloff	42,766
James A. LaBarre	28,632	Michael G. Savage	32,596		
E. Joseph Gess	28,510	Gerald F. Swiss	30,113		
R. Danny Huntington	27,903	Charles F. Wieland III	33,096		



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I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

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